



IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of: :
Phuong V. Luu et al. : Examiner: Eric J. Hug
U.S. Serial No. 10/702,414 : Group Art Unit: 1731
Filed November 6, 2003 :
Docket No. 12376 (GP-01-24) :
For: ABSORBENT SHEET EXHIBITING :
RESISTANCE TO MOISTURE :
PENETRATION :

Mail Stop Amendment
Commissioner for Patents
P.O. Box 1450
Alexandria, Virginia 22313-1450

DECLARATION UNDER 37 C.F.R. §§1.131, 1.132

Sir:

I, PHUONG VAN LUU, declare as follows:

1. I am currently employed by Georgia-Pacific Corporation at its Neenah, Wisconsin research facility and have been employed by Georgia-Pacific or its predecessors, Fort James Corporation and James River Corporation since 1989. Since 1989 I have

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worked in the field of paper tissue and paper towel manufacture and I have been awarded more than twenty (20) United States Patents related to this field.

2. I am the first-named co-inventor of the subject matter of the above-noted patent application and make the statements contained in this *Declaration* upon personal knowledge.
3. I understand from Counsel that the claims pending in the above-noted patent application have been rejected as obvious over United States Patent No. 6,758,943 to *McConnell et al.* and that this patent is effective as a prior art reference as of December 27, 2001. The claimed subject matter of the above-noted patent application (referred to as the present invention) is summarized in Claim 1, appearing below:

1. A method of making an absorbent cellulosic web resistant to moisture penetration comprising:
 - (a) wetting at least one surface of the web with an aqueous dispersion including a wax and an emulsifier; and
 - (b) heating the web above the melting temperature of the wax to fuse the wax of the dispersion and to provide a hydrophobic surface on the web, the wax being disposed in the web so that the open interstitial microstructure between fibers in the web is substantially preserved and the web has a laterally hydrophobic surface which exhibits a moisture penetration delay of at least about 2 seconds as well as a contact angle with water of at least 50 degrees at one minute of contact time with the web.

4. The present invention was conceived and actually practiced prior to December 27, 2001 as evidenced by the attached notebook pages which are redacted copies of pages from notebooks which I keep in the regular course of my research. Pages 97, 98, 101 and 102 are copies of pages from a first notebook which bear dates prior to December 27, 2001. Pages 4 and 12 are copies of pages from a second notebook which bear dates also prior to December 27, 2001.
5. More specifically, it is seen on attached notebook page 97 that the process of the present invention is described along with products enumerated in Table 2 of the application as filed:

TABLE 2

Moisture Barrier Results and Impact on Sheet Properties

Dispersion	Solids Melting Temperature*	Results
MICHEM ® 48040M2 Microcrystalline wax	73-94° C.	Very Good Barrier
MICHEM ® Lube 110 Carnauba Wax	85-110° C.	Good Barrier
MICHEM ® Lube 160 Carnauba Wax	85° C.	Good Barrier
MICHEM ® 44730 Polyethylene (A)	105° C.	Poor Barrier
MICHEM ® 39235 Polyethylene (B)	139° C.	Poor Barrier
MICHEM ® 71646M Microcrystalline Wax	92° C.	Good Barrier, no impact on softness
MICHEM ® Lube 124 Synthetic Wax	68-101° C.	Good Barrier, untreated side also hydrophobic
MICHEM ® 35160 Polybutylene	—	Good Barrier, tends to be strong when wet
Fiberglass X 9 Polypropylene	—	Coating very stiff
MICHEM ® 43040 Epolene ® Modified	—	Good Barrier
Polypropylene MICHEM ® 59740	—	Good Barrier

*approximate values reported by manufacturer

These products were made by way of the process of the present invention prior to December 27, 2001. I also note that an important feature of the present invention is heating the web which is provided with wax and emulsifier to a temperature above the melting point of the wax. This is seen in Table 2 above, for example, where the emulsions with higher melting waxes exhibit poor barrier under the conditions employed.



6. On attached notebook pages 98, 101, 102, the apparatus of **Figure 7** of the above-noted patent application is shown, along with the products of **Figures 8-11**:

FIG. 7

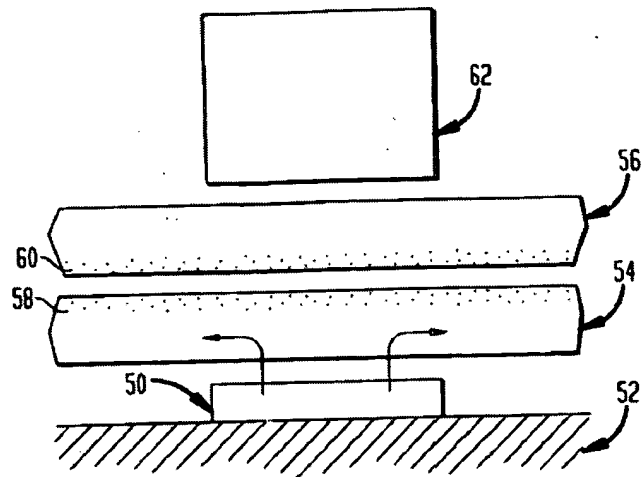


FIG. 8

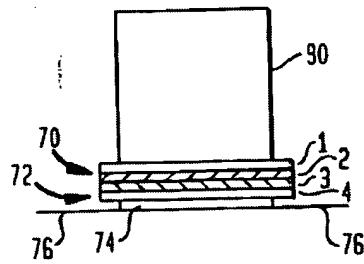


FIG. 9

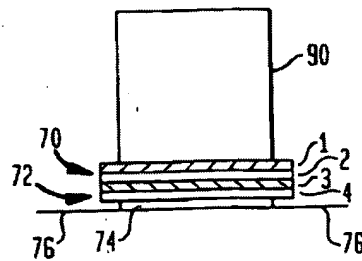
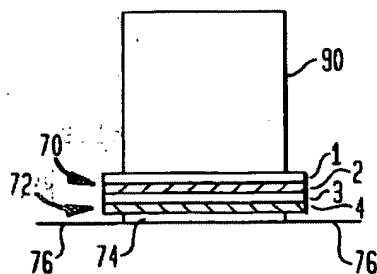
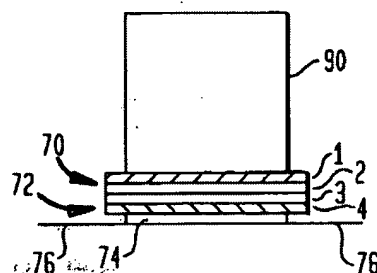


FIG. 10**FIG. 11**

Testing with the apparatus described on notebook pages 98, 101 and 102 was completed prior to December 27, 2001.

7. Attached notebook pages 4 and 12 describe further products prepared by way of the present invention prior to December 27, 2001.
8. Regarding the technical merits of the present invention, I am convinced that the present invention is unexpectedly effective at preventing moisture penetration as compared with the *McConnell et al.* '943 patent or any other reference of which I am aware. *Note* Table 3 and paragraph 153 of the application as published:

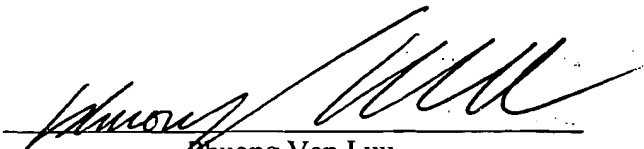
TABLE 3

		Wetted Surface Area (in cm ²) of Layers Under 1 PSI Pressure				
Towel Web Structure		Control	Example 14 (FIG. 8)	Example 15 (FIG. 9)	Example 16 (FIG. 10)	Example 17 (FIG. 11)
Top	Layer 1	17.2	0	0	8.3	0
Ply	Layer 2	17.2	0	14.9	8.3	19.1
Bottom	Layer 3	18.5	25	22.7	21.8	20
Ply	Layer 4	18.5	25	22.7	21.8	20

[0153] The multilayer structure exhibited an unexpectedly complete barrier to moisture penetration when the two treated surfaces of the towel were placed in contact with one another (Example 14). In all cases, the treated sheet exhibited resistance to moisture penetration and increased wetted areas in some plies over the control, suggesting migration of the emulsifier into the sheet.

9. The undersigned Declarant declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the subject application or any patent issuing thereon.

Signed this 07 day of November, 2006.


Phuong Van Luu

DATE

SUBJECT

 USING POLYETHYLENE, POLYPROPYLENE, POLYURETHAN.
 EMULSION FOR BARRIER TEST PROJECT NO. 170801209

97

It has been found that Polyethylene emulsion from Michelman Inc having following properties after hand-spray application and heating in the oven at 100°C:

1/ Delays the penetration of City water (or water created a barrier)

2/ Improve the surface hand feel of the sample

3/ Possibility thermobonding with another ply treated with Polyethylene emulsion

4/ Repulpable

The Polyethylene or polypropylene emulsion having Melting Point Temperature below 100°C is the refer chemical for this treatment application.

The following chemicals were used for lab evaluation.

1/ Michem Lubr 110. $T_m = 85-110^\circ\text{C}$ (Carnauba wax + Polyethylene) - good barrier

2/ Michem Lubr 160. $T_m = 85^\circ\text{C}$ (Carnauba wax) - good barrier

3/ MICHEMULSION 48040M. $T_m = 88^\circ\text{C}$ (Micro crystalline wax) - very good barrier

4/ MICHEMULSION 49780. $T_m = 105^\circ\text{C}$ (Polyethylene emulsion) - negative

5/ MICHEMULSION 39235. $T_m = 139^\circ\text{C}$ (Polyethylene emulsion) - negative

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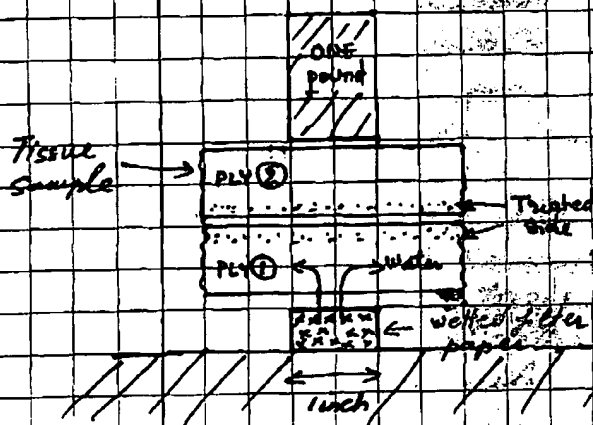
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Nichem emulsion 48040M2 is used to spray on the back side of

- 1/ Braumy with Twenty 0's
- 2/ Air-laid Essence Emmer Napkins
- 3/ Facial tissue Chelsea

The amount application is about 5% and after spraying the treated sample was dried in the oven at 100°C for 3 minutes.

The following test methods had been developed to characterize the water barrier property of paper samples.



- ① Filtered paper (1x1 inch) wetted with distilled water containing 1% NaCl.

About 0.8g (10 drops) for high BW sample

About 0.17g (5 drops) for low BW such as facial tissue.

- ② Put the paper sample on the top of wetted filter under a 1 lb pressure weight. The contact surface area of the wet should be 1x1 inch.

* The treated side either in contact with the wetted filter or in side. Both cases the contact time is 10 seconds for high BW and 5 seconds for low BW.

The results of Braumy towel, Air-laid web and facial tissue with non-treated side contacted to the wetted filter showed that there was no water penetrated inside the ply no 2 after testing.

In the case of Air-laid sample, water dispersion in the treated ply no 1 is significant faster than the non treated sample. This is indicated that the surfactant of Nichem emulsion was migrated in the web to improve the hydrophilic property of cellulose fibers and Polyethylene remained on the surface of the web to create a hydrophobic water barrier.

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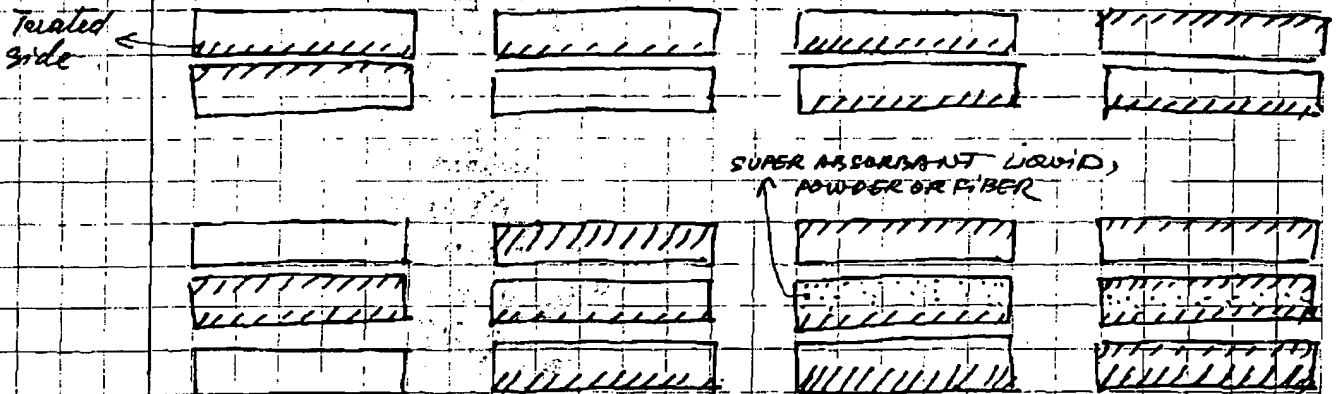
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SUBJECT Water & Grease Barrier Product PROJECT NO. 170801209 ¹⁰¹

The new products proposal:

- 1/ Bath tissue with Water Barrier (WB)
- 2/ Bath Tissue With applicator (Water or liquid lotion) treated (WB) or in using can be one side dry and other side is wet.
- 3/ Towel with WB or GB (Grease barrier)
- 4/ Towel with WB and GB.
- 5/ Facial tissue with WB
- 6/ Napkin with WB and GB
- 7/ Air-laid with WB and/or GB.
- 8/ Diaper - Feminine Pad without Nonwoven and without PE film cover

The barrier chemical may applied multiple with variable add-on level and may be design as below in the product structure:



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SUBJECT *Barrier Products*PROJECT NO. *170801209***PATENT CASE #2376
(FRAME-WORK)****A/ MATERIALS:****I. ACTIVE INGREDIENTS:**

- 1/ Water Barrier:
 - 1.1/ PE, PP, PD, PU, PS Emulsion.
 - 1.2/ PE-Co-Ac Na, PE-Co-MeAc Na Soluble in Water
- 2/ Grease Barrier:
 - 2.1/ PVOH
 - 2.2/ Poly(2-ethyl-2-oxazoline)
 - 2.3/ Fluorochemicals

II. BLEND ADDITIVES:

- 1/ Softener:
 - 1.1/ Surfactants: Anionic, cationic, nonionic, and amphoteric
 - 1.2/ Lotion emulsions
 - 1.3/ Emollients:
 - Natural: Avocado, tea tree oil, olive...
 - Synthetic: Mineral oil, alkyl benzoate ester, silicone
- 2/ Anti-bacterial Agent:
 - Organic: Triclosan, BAC, BEC
 - Inorganic: Zn salt, Cu salt, Ag salt

III. BINDER: PVAC, PAC, PSB, PA, and hot-melt (water soluble or insoluble)**IV. CROSSLINKER FOR PE-Co-Ac Na:** AZC, KZC, Zn organic salt.**V. SUPERABSORBANT:** Liquid or powder.**B/ PROCESS APPLICATION:****I. AT PAPER MACHINE:**

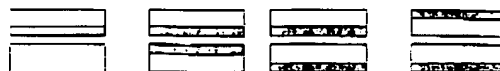
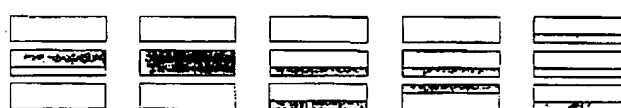
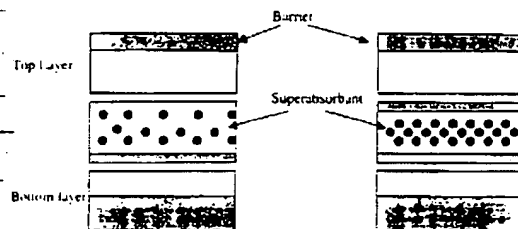
- 1/ Spray application *after Pressure Roll*
- 2/ Spray application *before creping blade*
- 3/ Spray application *after creping blade* (1 - 3 is CWP or TAD machine)
- 4/ Spray application *before drive roll* (on wet cast machine)
- 5/ Spray application *between two TAD*
- 6/ Spray application *before TAD of YTAD machine*

II. AT CONVERTING LINE:

- 1/ Before or after molding
- 2/ Curing at $>120^{\circ}\text{C}$

III. AT AIR LAID DRY FORMING MACHINE**IV. APPLICATION METHODS:**

- 1/ Spraying: Weko Unit, air, hydraulic, Super Critical CO₂...
- 2/ Coating: Curtain coater, printing, roll coating...

V. WEB STRUCTURE:**1/ One-ply:****2/ Two-ply:****3/ Three-ply Tissue, Facial Tissue, Napkin and Towel****4/ Three ply Air laid Diaper, Feminine Pad without Non-woven****C/ PRODUCTS:**

- 1/ Bath tissue
- 2/ Facial tissue
- 3/ Towel
- 4/ Napkin
- 5/ Air-laid napkin, towel...
- 6/ Diaper, incontinent, feminine pad without non-woven
- 7/ Wet & Dry tissue and towel with applicator
- 8/ Disposable garment
- 9/ Food contact product - Meat pads
- 10/ House wrap
- 11/ Disposable moist wiper
- 12/ Cosmetic cleansing pad
- 13/ Disposable clothing

D/ PRODUCT PROPERTIES:

- 1/ Water and/or grease barrier property.
- 2/ Bacterial barrier without using antibacterial agent.
- 3/ May improve base sheet softness, wet strength, and wet resilient and reducing product slickness.
- 4/ Reducing the discomforted feeling due to a very low amount of water or grease on the treated surface in using (diaper, feminine pad) or after wiping (napkin, tissue, facial tissue, towel product).
- 5/ Breathable
- 6/ Repulpable
- 7/ Dispersible

E/ TEST METHODS TO SUPPORT PATENT EXAMPLES AND PRODUCT CLAIMS:

- 1/ Absorbency Tester ATS-600 to measure absorption capacity VS time
- 2/ Microbial barrier testing with *Staphylococcus aureus* and *E. coli* bacteria
- 3/ FTIR reflection spectrum to characterize Z direction distribution of barrier chemical in paper web
- 4/ Soxhlet extraction to quantify add on level of the barrier chemical.

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Brownie 2 ply product is sprayed with Wickem Emul 4800042, then dried in the oven for 3 min at 105°C . Add on level of Barrier chemical are 2%, 4%, 8% and 11% by weight.

* The Air Perm Rate of these Samples:

Ref	Air Perm Rate (Sec/100 cm ²)
Control	0.5
2% treated	0.5
4% treated	0.5
8% treated	0.5
11% treated	0.5

* The SAT slow Rate Capacity Result.

Ref.	Time (Sec)	Rate (g/15")	Capacity (g/m ²)
Control	134 ± 35	0.1023	321 ± 11
4% Treated	886 ± 35	0.0377	310 ± 9
11% Treated (non-treated side down)	857 ± 659	0.0083	80 ± 80

The results show that:

* Air Perm unchanged when add-on level.

* The Capacity (g/m²) identical but time of treated samples (4%) to absorb is about 7 time longer than the Control.

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SUBJECT

WATER + GREASE BARRIER

PROJECT NO. 1708012C

1/ Mixed: 9g (40%) MICROM EMULSION 4048-M2 (Water based)
1g (20.25%) VININGS POLYGRAPHIX 700, contains
2.5% Fluorine as emulsifier, this is
a cationic emulsion material.

There is no phase separation.

2/ Mixed: 9g MICROM EMULSION 4048-M2.

1g (18.33%) ZONYL NF lot 195, this is
anionic material.

There is no phase separation.

Gray ~~2~~ formulation ① or ② on the brown
level with add-on rate about 3-5%. Then
dry the wetted sample in the oven for about
5 min at 100°C.

The sample treated with both formulation ① or
② were performed high grease barrier ^{properties} when
testing with soybean oil. The delay time of oil
penetration is > 80 sec. However, the non bio
side was not delayed absorption of grease.

The water barrier of both formulation is appear
performing delay water penetration similar to the
formulation without grease barrier chemical.

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